

INDIAN GRAPHITE

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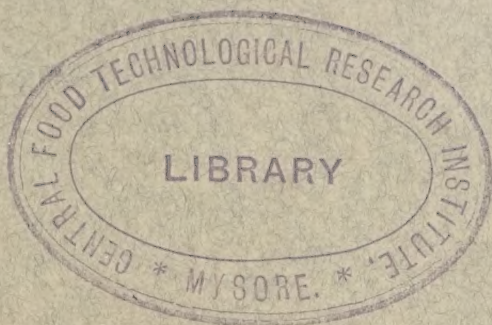
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C. S. I. R. MONOGRAPH



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ITS BENEFICIATION AND PROBABLE USES

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SIR J. C. GHOSH,
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I GRAPHITE

Graphite and diamond are the two crystalline allotropic forms of the element carbon, while charcoal, coke, gas, carbon etc., are the amorphous allotropes ; but recent studies (1) indicate that even the amorphous varieties of carbon consist of a "series of physical states without visible crystallisation and with a minimum density in more or less crystallised carbon and with a maximum density in the well crystallised graphite." Graphite can be manufactured by heating coke to a very high temperature in an electric furnace. The artificial graphite so obtained is very finely divided and extremely pure.

Natural graphites are usually divided into three classes, the flaky, the crystalline and the amorphous*, depending upon the mode of occurrence. The flaky variety is the scaly crystalline form of the mineral, commonly found disseminated in metamorphic rocks such as crystalline limestones, gneisses and schists. Each flake is a separate individual and has crystallised as such in the rock. The crystalline graphite on the other hand is found in the form of more or less well defined veins or as accumulations in pockets along the intrusive contacts of pegmatites with limestones, schists, etc. Both the flaky and the crystalline varieties represent fissure or fracture filling minerals but differ only in external appearance and in their mineral impurities. 'Amorphous graphite*' is usually found in the form of minute particles more or less uniformly distributed in feebly metamorphic rocks such as slates and shales. They are either metamorphosed coal seams, the graphite content of which goes up to 80-85 percent or altered carbonaceous sediments having 12-60 per cent graphitic carbon. In addition to the varieties described above Spence (2) finds it more convenient to employ the term 'Vein graphite' to describe those pocketed bodies of crystalline graphite, which are characterised by their irregular form and the abundance of inter-grown foreign mineral substance. Many varieties of Indian graphite can be placed under this class.

There is a considerable difference in opinion regarding the origin of graphite (3), (4). It is generally believed that graphite is formed by different processes by magmatic separation of the mineral as an original constituent or as the result of the assimilation of carbonaceous rocks by

*The term 'amorphous' is a misnomer since all varieties of graphite are crystalline.

pneumatolytic action or by the metamorphism of sedimentary rocks that contained original carbonaceous matter.

Graphite crystallises in the hexagonal system often in the form of scales or plates or in large foliated masses. It has a perfect basal cleavage. Its hardness varies from 1 to 2 and it feels greasy to touch. The cleavage flakes are perfectly opaque and are flexible but not elastic. Its specific gravity ranges from 2.1 to 2.3. The purest Ceylon graphite has a specific gravity of 2.25. The crystalline flakes of graphite have a black to steel grey metallic lustre. The amorphous variety however is matte, black and earthy in appearance. Graphite is a good conductor of heat and electricity. Its thermal conductivity (in — 100 mesh powder) is 50-75 B.T.U./hr./sq. ft. °F per foot. The specific heat of graphite is 0.2019 and is found to increase considerably with temperature. The calorific value is found to be 7779.45 cal./gm. The temperature of fusibility is unknown but is probably above 3000°C.

Natural graphites are never found pure, but are invariably associated with
 Chemical foreign minerals, like clay, calcite, quartz, iron oxide and mica.
 Properties

Graphite is highly resistant to weathering influences and to chemicals. Fusion with alkali carbonates produces carbon monoxide. Graphite is not affected either by hydrochloric acid or by hydrofluoric acid but yields graphitic acid on treatment with a mixture of potassium chlorate and nitric acid. It is combustible in presence of oxygen above 620°C, but is unaffected even at 2000°C in an inert atmosphere. Flaky graphite is more resistant towards atmospheric oxidation than the other varieties and hence is largely employed in the manufacture of graphite crucibles. Mantell (5) and Donath (6) have given the details of the properties and the manufacture of graphite and graphite articles.

By virtue of its refractoriness, its capacity to conduct heat and electricity,
 its chemical inertness, its softness and lubricating properties,
 Uses of Graphite graphite finds a variety of uses in science and industry.

The major portion of the world's flaky and vein graphite is employed in the production of graphite crucibles retorts, muffles, saggars, pouring nozzles, skimmers, crucible covers and other articles designed to withstand high temperatures (5), (7). These articles are generally manufactured by pressing a mixture of graphite, clay and sand and firing the pressed articles to a high temperature to introduce the required hardness and strength. By virtue of its heat conduction, graphite imparts resistance to sudden changes in temperature. The manufacture of these refractories requires good quality of flaky graphite of 20-100 mesh size.

Prior to the introduction of the technique of artificial graphitisation of carbon, natural graphite did find a place in the manufacture of electrodes. Natural graphite is still employed as an additional ingredient in the manufacture of certain types of carbon brushes for motors and generators (5), electrical contacts, rheostats, plates, discs, rods and electrical plungers

The lustrous, greasy, chemically inert and water repellent nature of flaky graphite makes it an ideal substance in the paint and pigment industry (5) (8). When mixed with inert substances like silica, the quality of the graphite paint improves and this is largely employed as a steel paint. Red lead and aluminium paints are found to last longer when they are mixed with graphite. The water repellent action of graphite makes it an ideal protective coating in the preservation of wood. Graphite paint is of particular importance in the alkali industry where most of the paints get corroded. Finely powdered crystalline graphite (70% pure) is generally employed in the paint industry.

Graphite is the most important material used for foundry facings. It prevents the sand of the moulds from adhering to the metals that are being cast (9). The best results are obtained when high grade flake graphite is used; but low grade dust product from refining mills containing 40-70 per cent. graphite is also widely employed. Large quantities of amorphous graphite too poor in quality to be used for other purposes are also utilised in foundry facings (5). Foundry facings account for the production of a large proportion of the natural graphite.

Another important use of graphite consists in the manufacture of pencils (5), (10). The soft amorphous varieties are best suited for this purpose although finely divided flaky varieties are also frequently used. It is estimated that about 8% of the world's production of graphite is consumed in the manufacture of pencils.

Graphite is the most important solid lubricant and can be used either with oil or with water. Acheson gave the terms 'Oildag' and 'Aquadag' for the suspensions of colloidal graphite in oil and in water, respectively. By virtue of its fine state of division, artificial graphite is invariably employed in the manufacture of lubricants. During the war period the supply of graphite lubricants was very inadequate and at the instance of the Supply Department, Government of India, preparation of graphite lubricants from naturally occurring Indian graphite was undertaken at the Indian

Institute of Science, Bangalore. The details of the methods employed for this purpose are given in a later section. Graphite greases are still very popular as heavy duty lubricants where high temperatures or washing action may tend to remove the grease.

Graphite has several minor uses (5). Finely divided graphite is used in Miscellaneous Uses electrotyping where its electrical conductivity is taken advantage of. A high grade variety of graphite is required for this purpose. Amorphous natural graphites are used in making stove-polishes (5). Graphite is used to impart a protective moisture-repellent finish to powder grains. It is also used in polishing gun shots. Graphite is used in belt and cable dressings. It is employed in engine packing hard rubber compositions, cord and twine manufacture. Various types of washers and packings are made with graphite powders. Mixed with lead it forms one of the best high pressure corrosion resistant packings (5). Graphite is often used as a filler in fertilisers to prevent moisture absorption and subsequent caking. It is sometimes added to magnesia-brick ingredients to assist moulding and to increase the heat conductivity. Graphite is also employed in boilers to prevent scale formation. The finely divided graphite works its way through cracks in the old scale and loosens up the whole mass.

II OCCURRENCE OF GRAPHITE IN INDIA

Although graphite occurs in various places in India, regular prospecting work has not been undertaken so far and it is not possible to give an estimate of the quantity of the mineral available at various places. Workable deposits are confined to metamorphic and igneous rocks where graphite occurs in pockets and veins. The following extract from an article by M.S. Krishnan (11) gives an idea of the various deposits that are located in India.

A vein of graphite with an average thickness of 13" occurs about half a mile to the north of the Tsuntang-Lachen road. The composition of the bulk samples is reported to be 93% graphite (12). Certain graphite schists of low carbon content are also said to occur in the Darjeeling District (13). The economics of the working of these deposits have not been examined.

Graphite is mined near the Kumandih and Palamau districts and is used for the foundry facing by the Bengal Iron Work at Kulti. The graphite content of these samples is 35% (11). Flaky graphite is also reported to occur in the Palamau district but the graphite content is too

poor for working these deposits (14). Graphite deposits are also located in Dimadiha and Bagmari (11) but the details are not known.

Extensive deposits of graphite are said to exist 3 miles north of Betul. The Central Provinces graphite is mined from this area and the concentrates contain about 35% or more of graphite.

A number of graphite deposits have been discovered in the Patna State, Eastern States Agency the more important of these being located in the sub-divisions of Titilagarh, Bolangir and Patna. A beneficiation plant and a crucible factory are working at Titilagarh. Nearly sixty different deposits are reported to have been located in the Patna State and many of them are being worked since 1937 (11). After preliminary cleaning, washing and grading, the samples are utilised for the manufacture of crucibles by 'The mining and Allied Industry, Patna State, Ltd. Messrs. 'Patna State Graphite Mining Co.' are the chief exporters for the Patna State graphites. The flaky graphite deposits of Bastar State are not considered to be of economic importance (15). Several graphite bands with widths varying from 18 inches to 9 ft. having 40-65% graphite are found to occur in the Kalahandi State but their economic importance is not yet fully investigated. The Indian Graphite Co. was mining graphite near Kasurpara during the 1914-18 war but the mining is now discontinued. Some pockets and veins of graphite are also located near Santpur and these are known as Koladighat deposits (16). The utility of many of these deposits has not been fully investigated.

During the past few years graphite is being mined in the Paloncha Samasthanam in the Warangal District. The mineral contains 60-90% of graphite. Hyderabad State

Abundant quantities of amorphous graphite have been known to occur at various places in Kashmir State. The biggest band is situated in the Braripura area of Uri Tehsil and extends over 4-7 miles in length and 400 ft. in thickness. Three large deposits have been investigated in detail in this area (17). The central deposits near the Braripura area are estimated to yield 137,500 tons of purified graphite in addition to 4 million tons of second grade graphite. The western and eastern deposits are known to yield about 800,000 and 350,000 tons respectively of a poor quality (22%) graphite. These large deposits are worth investigating. A few deposits known to occur in the N.W.F. Province are all too poor in quality and are not of any economic importance.

Graphite occurs in certain areas of East Godavari District as veins extending over 50 ft. in length 5-6 ft. in width and about 2 ft. in thickness. The mineral is also reported to occur in several other neighbouring places like Valegalapalle, Marinpalem and Kothada Village (11). A good quality of flaky graphite is being mined near Zangareddigudem, West Godavari District. This deposit is estimated to yield at least a few hundred tons of the mineral. The following are some of the places where graphite is reported to occur:—(1) Kurinjakulan of the Tinnevely District where several hundreds of tons of flaky graphite (5-10%) are found; (2) Peddamadur of the Kistna District (18) where abundant spangles of graphite are found and Narasapattam of the Vizagapatam District where the mineral was being mined during 1910-1911.

Fine grained amorphous graphite mixed with a large quantity of clayey matter (about 90%) is found at Ganacharpura, Kolar District, and is supplied to the Mysore Iron & Steel Works for the foundry facings. Two more deposits of flaky graphite are reported near Mavinahalli and Sargur in the Mysore District. The Mavinahalli deposit is estimated to yield 3500 tons of 10% graphite (19) while the Sargur deposit although extensive is too poor in quality (2-3% graphite) to be of any economic importance (20).

Graphite is found in a number of localities in Travancore the details of which can be had from the reports of the State Geologist for the years 1907-1911. The deposits are located near Amanad, Arumanallur, Attungal, Avaneeswaram, Mamalai, Pathanapuram, Kolachel, Punalur and Vellanad. The Morgan Crucible Company worked some of the deposits near Kolachel (11) for some years in the early parts of the century and were producing 2000-3000 tons per year.

Graphite has been observed in a band of schists near Sohna in Gurgaon District. Some specimens from this area show 78.5% graphite. Extensive deposits of graphite shales are reported to occur in the Lesser Himalayas, especially in the Mandi and Suket States of the Simla Himalayas (11).

A poor quality graphite bed is located near Bar railway station in the Jodhpur State. Graphite bands are also noticed near Rajore, Lotiana (Ajmere-Merwara district), Ajitgarh and Cheriabarb (11). The graphite mined at Kishangarh is used for the manufacture of black paint (11). Beds of graphite shale are also found near Kalighata, Jawar and Udaisagar but no details of prospecting are available.

Small segregations and pockets of graphite are reported near Almora, United Provinces, Pulsimi and Dol (11), but the details of prospecting are not available.

Good quality flaky graphite is reported to occur in the ruby-bearing limestone beds of Naniazeik (22) and Wabyudaung and Kyaukgyi. Burma and Ceylon The vein graphite deposits at Taw-Yokyaung are not found to be of economic importance (23). Black graphite schists have been noticed at Pagaye, Yeu, Toungoo and Kamaing (24). Graphite deposits of Ceylon are found to occur in three main groups in the south-west part of the island, Regedera group in the North, Kulatura group in the centre and Hambantota in the south. About a dozen mines are being worked on a large scale and the annual production ranges up to 30,000 tons. It has to be pointed out that some of the purest varieties of flaky graphite are obtained from Ceylon.

III PRODUCTION, IMPORTS AND EXPORTS OF GRAPHITE

Dunn (25) has estimated that the total annual output of graphite in the world is about 120,000 to 150,000 tons and a considerable proportion of this quantity is produced artificially in the electric furnace. But as pointed out previously natural graphite is required for certain special purposes like the manufacture of crucibles, retorts, muffles etc. The accompanying table (I) compiled from the 'Statistics of British India' and the 'Records of Geological Survey of India' gives the production of graphite in India.

TABLE I
Production of graphite in India in Tons

Locality	YEARS					
	1901-1912	1915-1922	1929-1935	1936	1937	1938
Eastern States	...	597	228	438
Travancore	23873
Central Provinces	...	47	505	20
Godavari	991
Rajputana	...	1186	39
Mysore	...	20	388	...	320	...
Total Production :						
Average per year	2072	231	129	...	548	458

Reference to table I indicates that the production of graphite in India is almost negligible when compared with the annual production of the world (viz. 150,000 tons). During the war period the production of Indian graphite might have increased to about 2000 tons per year. Even this figure is too small when compared with the production of graphite in Ceylon (30,000 tons in 1942).

During the early period of the present century India had an export trade in graphite up to 1913. World War I stopped all exports and since then the export trade has never revived.

The following table gives an idea of the import of graphite and graphite crucibles during recent years.

TABLE II
Imports of graphite and graphite crucibles

Year	Graphite in (tons)	Graphite crucibles in (tons)	Value of Graphite (Rs.)	Value of gra- phite crucibles (Rs.)
1919	161	146	79,090	90,270
1920	410	177	151,100	192,600
1921	422	47	214,349	70,393
1929	511	180
1930	495	153
1931	502	203
1937	528	174	131,436	162,829
1938	555	273	155,722	243,690
1939	419	157	113,902	155,077
1943*	71	225	65,318	360,025
1944*	38	328	34,971	494,049
1945*	455	192	241,390	294,930

*These figures are for 8 months only.

In this connection it may be of interest to know the amount of lead pencils imported into India. In the year 1937, India imported 5600,000 dozens of pencils valued at Rs. 10,72,000. A large part of this came from Germany and Japan (10). This gives a rough idea of India's annual requirements, which are expected to increase in proportion to India's educational advancement.

The cost of mining in India is estimated by Dunn (26) to be Rs. 3/4 per ton. The cost of purification may vary between Rs. 30/- and Rs. 40/- per ton of the concentrate depending upon the efficiency of the method of purification. Thus the net cost of washed graphite will be roughly Rs. 50/- per ton. Prior to the war good quality graphite was being sold at Rs. 50-100/- at the mines (27).

According to the "Statistical Abstract for British India with Statistics where available relating to certain Indian States", in 1937 the production of graphite from Madras was valued at Rs. 100/- per ton, that from Mysore at Rs. 20/- per ton and graphite from East Godavari at Rs. 39/- per ton. In 1938 graphite from C.P. was valued at Rs. 49/- per ton and from Eastern states at Rs. 45/- per ton.

Prior to the war one ton of imported graphite would cost Rs. 250-270/- for the years 1936-39 (28). Godbole (10) gives a figure of Rs. 300/- per ton of the imported graphite at ports.

The war years however witnessed a considerable rise in the price of graphite. In 1943, a firm in Travancore quoted Rs. 180/-, 220/- and Rs. 500/- per ton of different grades of graphite. In the same year a Madras firm quote Rs. 900/- per ton for graphite of good quality. An abnormal figure of Rs. 2240/- and even Rs. 3360/- per ton was quoted by a firm in South India for a very fine variety of flaky graphite. Most of these rates however were fantastic since imported graphite would cost only Rs. 920/- during 1943-44 (Table II).

V REFINING OF GRAPHITE

The chief impurities in natural flaky graphites are mica, calcite, quartz, feldspar, iron sulphide and silicates of calcium, magnesium and aluminium. In cases of pronounced weathering, many of these impurities are converted into clayey matter often resulting in considerable enrichment due to natural leaching. The graphite content of a fresh flaky ore varies between 10-30% while the soft decomposed ore often contains 30-50% graphite. The crystalline graphites are the purest of all natural varieties, the Ceylon sample being the purest in the world. Some selected Ceylon crystalline samples contain over 99% graphite. Hence for a long time Ceylon held a dominant position in the world's production of

crystalline graphite used for the manufacture of crucibles. Recently the flaky graphitic ores coupled with the improvements in the methods of refining have competed successfully with the Ceylon crystalline graphite. "Amorphous" graphite ores generally have a low graphite content with the exception of the altered coal beds of Mexico. This grade has been in special demand for the manufacture of lead pencils. The lower grades of amorphous graphite are very difficult to refine owing to the extreme fineness of the particles. Such varieties are generally employed in the construction of foundry facings without any purification. The importance of the naturally occurring amorphous graphitic ores has been considerably diminished after the advent of artificial graphite.

As in the case of other minerals, transport, fuel, general economic policies, the state of industrialisation, etc., play a vital part in determining the importance of graphite deposits.

Methods of Refining

From the beginning of the history of utilisation of graphite, various Preliminary Treatment methods of refining both dry and wet have been employed with varying degrees of success. Extremely pure ores like the 'Plumbago' from Ceylon do not require any complex treatment. The mineral is hand-picked and sized, the larger lumps being sold as 'Lump' graphite while the smaller sizes go as 'Chip' and 'Dust'. The lower grade material is generally pulverised and sometimes washed with water, dried, winnowed and graded into 'Dust' and 'Flying dust'. The amorphous variety varies so widely in composition and in physical condition that the treatment depends upon the use to which it can be put. In the case of high grade ores containing above 80% of graphite, the material is just crushed and used while slightly inferior ores are generally ground to very fine sizes (200-350 mesh per inch) and then air floated. There seems to be very little information regarding other methods of beneficiation of amorphous graphite.

A number of dry methods are employed in the concentration of Dry Methods of Concentration graphitic ores. Separation of gangue from graphite can be effected by means of pneumatic tables or jigs. Elimination of the brittle gangue constituents from the graphite is carried out by successive grinding and sieving. This method takes advantage of the difference between the grindability of the gangue and the graphite particles. Air classification can also be employed for the concentration of the powdered mineral. Separation of

the graphite from the gangue can also be effected by electrostatic methods by passing the pulverised ore through a strong electric field when the graphite flakes are deflected from the path of the gangue material. Although the dry methods can be employed for the beneficiation of graphite, yet they are comparatively less efficient and more expensive. Hence the application of these methods on a commercial scale is impracticable in the case of graphitic ores.

Many chemical methods have been tried to effect the purification of graphite. In these methods, the gangue material is brought to a soluble condition by treatment with drastic reagents like concentrated sulphuric acid, potassium chlorate, hydrofluoric acid, fused sodium carbonate and the unaffected graphite is separated by filtration. But these methods are very tedious, highly expensive and can never be employed for the industrial beneficiation of graphite.

Among the wet methods of refining, treatment with buddles, log washers, rake washers and wet tables have been employed for washing the graphitic ores. Washing can be of advantage if the gangue contains clayey and finely dispersed impurities, and when this is not the case washing has very little effect. Mere tabling has not been widely applied in the beneficiation of graphite because of the small difference in specific gravity between the graphite and the associated gangue material. All the methods outlined above have, according to modern standards failed to give satisfactory results in the beneficiation of graphite when graphite occurs in finely divided condition associated with gangue material.

Beneficiation by 'Froth Flotation'

The new process that has been evolved to separate finely divided graphite from the gangue is known as the 'froth flotation' process. In this process graphite is separated from the clayey matter by taking advantage of the sticking property of the graphite to an air bubble when air is blown through a suspension of the pulverised graphite ore in water. The clayey material does not stick to the air bubble so firmly and hence the separation is effected. Before going into the details of the process it will be of interest to know the development of this process as applied to graphite.

In 1877 Bessel and Bessel took out a patent for separating graphite from clayey matter by mixing the pulverised ore with 1-10% of paraffin oil and boiling the mixture with water. The steam bubbles that carried most of the graphite particles were collected and the

graphite separated (30). Later (31) a second patent was taken out in 1886 to evolve a gas in the suspension of the ore in water by the action of an acid on carbonates. The modern froth flotation process employs the same principle as that of Bessels but differs in details such as the use of the oil, the method of production of gas in the aqueous suspension of the ore, etc. The work of Bessels was soon lost sight of and the modern process was finally developed as the result of coordination of the work of several investigators. In 1898 Elmore developed a process for the beneficiation of 50 tons of copper ore by the flotation method (32). In this process a large quantity of oil was used for the treatment of the ore. The first successful commercial plant was invented by Potter (33) who beneficiated six million tons of zinc carbonate ore from an initial concentration of 20% to a final concentration of 42%. The process of Froment (34) employed a small percentage 1-1.5% of oil for the flotation process. In 1904 Cattermole discovered the use of soaps, acids and alkalis in the flotation process (35). Since then a lot of work has been done on the different aspects of froth flotation and today the process is employed for the beneficiation of a large number of minerals. Taggart's book on ore dressing (36) gives full details about the various machinery employed in froth flotation.

The details of the principles involved in froth flotation have been described by a number of investigators (37), (38), (39), (40), (41). In this section attention is restricted to the fundamental principles involved in the application of froth flotation, as applied to graphitic ores. As pointed out previously when air is bubbled through an aqueous suspension of finely divided gangue and graphite, the air has a natural tendency* to drag with it a large number of graphite particles and only a small number of gangue particles. But when the bubble bursts at the surface, the particles carried by the gas bubble will again sink to the bottom. It is thus obvious that if we want the graphite particles to remain with the gas bubble at the surface, the bubble

*Note:—There seems to be some difference in opinion regarding the natural attraction (floatability) between graphite and air. Gaudin (42) believes in the natural floatability of graphitic ores while Wark and Cox (43) have shown that in presence of certain frothers graphite does not float without the addition of collectors. They have also shown that the frothing agent employed may also function as a collector. Del Giudice (44) states that all well polished minerals exhibit a zero contact angle (no natural floatability) and infers that graphite also obeys the same rule. In his recent book (45) Gaudin states that the natural floatability of graphite may be due to the contamination by organic substances. Experiments carried out by J.C. Ghosh and R. Banerjee at the Indian Institute of Science have shown that even the purest Ceylon sample (ash content 0.3%) of graphite gives rise to a contact angle of 23° when immersed under distilled water. But on continued washing with distilled water, the contact angle is reduced to zero. The contact angle increases to 50° when the sample is treated with ethyl xanthate solution. Hence it can be concluded that graphite as it occurs in nature exhibits a contact angle (attraction towards air) although this might be due to the presence of impurities.

should not be allowed to burst when it reaches the surface. In other words, a stable froth must be produced when the gas is bubbled through the aqueous suspension. The reagents that are added to water to produce stable froths are known as frothing agents or frothers. The following are some of the common frothers employed in the flotation of graphite:— (1) pine oil (2) cresylic acid, (3) eucalyptus oil, (4) camphor oils, (5) certain alcohols. Sometimes a combination of two or more of the frothers is employed. After producing a stable froth, the next thing of importance is to increase the attraction between graphite and the air bubble so as to prevent the former from coming down. This is generally effected by adding certain reagents known as collectors. Some of the common collectors that are employed in the beneficiation of graphite are (1) kerosine oil, (2) cresylic acid, (3) coal tar oils, (4) xanthates, and (5) aerofloats.

So far, attention has been devoted to increasing the attraction between graphite particles and air bubbles. As stated already, the air bubble can also carry a small number of gangue particles and it is essential to diminish the attraction between the gangue and the air bubble and thus minimise the quantity of the gangue carried by the air bubble. The reagents used for this purpose are known as depressants since they depress the attraction between the air bubble and gangue. The reagents generally employed for this purpose are:— (1) sodium silicate, (2) glue, (3) gelatin, (4) gum, and (5) sodium cyanide. Amongst the other factors which influence the carrying power of graphite by the air bubble the following are the more important ones:— (1) acidity of the aqueous suspension, (2) particles size of the suspension, (3) percentage of solids present in the suspension. All these factors have to be carefully controlled to have the maximum efficiency in the beneficiation of graphite. The following are the details of the several processes employed in the flotation of graphite.

Good frothers are organic substances of a complex nature composed of two groups, one being polar (hydrophilic and the other non-polar (hydrophobic). The common examples for the polar group are: COOH , OH , CO , NH_2 and CH : the non-polar group forms the rest of the organic molecule. The stability of a froth is brought about by the lowering of surface tension of water and also by the increase in the surface concentration of the frother (46). The size, the stability and the toughness of the bubbles composing the froth are all of primary importance. If the size of the bubbles of the froth is very small, the drainage of the gangue material is interfered with. If the bubbles are too large, the effective area of the froth

bubbles will be small and consequently flotation will be less efficient. Hence the optimum size of the bubbles must be determined for each mineral. The toughness of the froth is equally important. If the froth is not tough, the graphite particles tend to fall back while too much of toughness involves mechanical problems in subsequent operations. Alcoholic frothers generally produce soft or brittle froths while coal tar and creosotes produce tough froths. The quantity of frother required varies from 0.05 to 0.20 lbs. per ton of the ore treated. Overdose of the frother should always be avoided since it destroys the froth. The character of the froth is also determined by the nature of the aeration in the flotation machine. Effectiveness is increased by the production of finely divided air bubbles distributed evenly throughout the entire pulp.

The purpose of a collector in the flotation process is to render the graphite highly water repellent so as to effect an efficient separation of graphite from the gangue. Without this aid froth flotation would be impracticable. Once it was thought that only oils or oily substances which were insoluble in water would act as effective collectors. But the discovery of modern soluble collectors like the xanthates and aerofloats (37) has disproved this idea. What is really important is that the mineral must adsorb the collector strongly; at the same time the gangue material should not adsorb the collector to any extent. As in the case of the frother, the collector molecule also should have the polar and the non-polar groups; the polar group attaches itself to the mineral while the non-polar orients itself towards the aqueous phase. Sometimes one and the same substance can function both as a collector and also as a frother. Cresylic acid is an example of this type of combined reagent. The quantity of the collector that is added ranges from 0.01 lb. to 2.5 lbs. per ton of the ore treated. A list of collectors and frothers employed in the flotation of graphite is given in table III of page 15.

The function of the depressant is to retard temporarily or permanently the flotation of the gangue material. This considerably improves the efficiency of the beneficiation process. The common inorganic depressants are lime, sodium cyanide (51) sodium sulphate and sodium silicate (50). Tannin, starch and glue are some of the organic depressants. The depressants are generally added to the grinding circuit or the conditioner before the addition of the collectors and frothers. The amount to be added has to be predetermined.

TABLE III

Substance	Employed by
1 Kerosine (collector)	Bessel Brothers in 1877
2 Brown coal tar oils (collector and frother)	Landgraebaer in 1923
3 Cresol, turpentine, tar and gasoline (collector and frother)	Elie. Brit. Patent 355211 (1929)
4 A mixture of kerosine and pine oil (frother)	Parsons in 1926 and also by a large number of workers in U.S.A. between 1926 and 1945 including Alabama Flaky Graphite Co.
5 Crude oil (collector) pine oil (frother)	Alabama Flaky Graphite Co. (1934) (1941)
6 Kerosine (collector) Pine oil (frother)	Workers in U.S.S.R. (1934)
7 Polythionates (collector)	W. Schaefer and Erz (1927)
8 Xanthates (collector)	Wark and Cox (1935)

Amongst the factors that regulate the carrying power of the collectors, the acidity of the pulp is very important. The acidity is generally expressed in terms of pH. The pH of the graphite suspension depends upon the nature of the collector employed. Thus when xanthates are employed, an alkaline medium (pH=8) should be maintained while for cresylic acid the medium should be acidic (pH=3-5). In the case of kerosine oil, however, the pH of the medium does not seem to have much influence on the collecting power. The suitability of the pH depends upon the nature of the gangue material also. Thus if the gangue consists of a basic material like lime, an alkaline medium should be preferred. But for neutral gangue like sand or clay one can employ both the acidic and the alkaline media.

The preliminary process of grinding the mineral to the requisite degree of fineness in order to ensure that the valuable minerals have been liberated to a satisfactory degree, is of great importance. On this depends both the recovery and the grade of the concentrate. After the preliminary crushing, in heavy crushers of the jaw crusher type, the ore is usually ground in ball or rod mills in presence of water so that a flowing pulp is obtained. The grinding is usually carried out in a closed circuit with classifiers which, not only control the extent of the size reduction, but deliver the graphite in a pulp of suitable density for flotation. In practice grinding is carried out to the most economic limit and complete liberation of the graphite from the ore need not always be the criterion.

Mineral particles between the sizes 48 and 500 mesh per inch can be recovered by flotation. Extremely coarse and extremely fine particles do not float well and the recovery is low. The optimum size distribution necessary for good recovery and grade has to be determined by laboratory tests. The usual range in practice lies between 50 and 250 per inch.

Gaudin (47) points out that in the flotation stage the pulp must be dilute enough to permit particle rearrangement and that no pulp thicker than 35-40% solids by weight can be used. In the conditioning stage the thickest kind of pulp is desirable as it results in the economy of reagents consumed. In the separating stage, particularly in the cleaning operations pulps of 15-20% solids or even less might be necessary. For the beneficiation of graphite a pulp density of 20-25% is found to be satisfactory in U.S.A.(52).

Wark draws attention to the fact that any change in a variable, which raises the grade of concentrate, simultaneously tends to lower the recovery. Thus the flotation engineer is generally faced with the problem of determining how best to reconcile the grade of the product with recovery. A balance can be attained only by a consideration of the industrial demand and the price commanded by the various grades of the mineral in question. Each ore thus gives rise to a specific problem and no general statements can be made to give a ready formula for any type of ore.

The standardisation of the flotation process involves thorough knowledge of the several physico-chemical properties of surfaces like surface tension, surface energy, contact angle, polarity, surface reactivity and adsorption. The success of the flotation of a sample of graphitic ore requires a careful control of these various factors to bring about the beneficiation with the utmost economy.

The pH of an aqueous solution is defined as the logarithm of the hydrogen ion concentration with a negative sign. Thus the hydrogen ion concentration of 0.1 N hydrochloric acid is 10^{-1} mole per litre while its $\text{pH} = -(-1) = 1$. Similarly the pH of water is 7 since the concentration of hydrogen ions in water is 10^{-7} mole per litre. The pH of 0.1 N sodium hydroxide can be shown to be 13 by similar reasoning.

V BENEFICIATION OF INDIAN GRAPHITES

During the war period the supply of graphite and graphite lubricants from abroad became scarce and the Supply Department suggested that beneficiation of graphite for the preparation of graphite lubricants should be investigated. The Council of Scientific and Industrial Research decided that this work should be carried out at the Indian Institute of Science, Bangalore * A number of representative samples from various parts of India were collected, analysed and the samples were subjected to beneficiation by the flotation process. In all these beneficiations attention was mainly devoted towards the preparation of as pure a sample of the graphite as possible since silicious impurities are highly objectionable in graphite lubricants.

Collectors

(1) Potassium ethyl xantate was prepared by the standard methods described by Foster (49) and Wark (37). The reagent was used without further purification. (2) Eucalyptus oil, cresylic acid, cresols, turpentine oil, kerosine and sodium silicate were purchased from the market and used without further purification.

The graphitic ore was first crushed in a small jaw crusher, then ground in an edgerunner and finally pulverised in the dry condition using a porcelain ball mill. Wark (37) has shown that wet grinding is not necessary in the case of graphitic ores. After pulverising, the samples were subjected to wet sieving. The sieved samples (1-1.5 lbs.) were then transferred to the laboratory flotation cell (the Ruth Sub-aeration type having a capacity of 3.5 litres) where it was mixed with about twice the weight of water, and the mixture well stirred by an automatic stirrer provided for the purpose. The pH regulators and the depressants were then added and the pulp agitated for about 2 minutes. The collector was next added and the pulp conditioned for 5-10 minutes. The pulp density during conditioning was maintained at 30-40% solid contents. Subsequently the frothing agent was added and the pulp diluted. Air was then allowed in to produce froth in the mixture. The mineralised froth formed on the surface of the cell was removed by means of wooden paddles and collected in a bucket. The concentrates were filtered, dried in an

*The experimental work described in this pamphlet was carried out by K. Mapindar and R. Banerjee under the guidance of Sir J.C. Ghosh at the Indian Institute of Science, Bangalore.

air oven and analysed. Preliminary experiments conducted on a small sample of the pulverised ore (10-15g) using a separating funnel as the flotation cell, indicated the optimum concentrations of the collector, frother regulator and depressant that had to be used.

In addition to the mechanically agitated laboratory flotation cell, a larger air lift cell was constructed according to the plan described by Rabone (53). The lower portion of the cell was V shaped. Air at a pressure of 1-2 lbs. per sq. inch was delivered from a blower to a header which ran longitudinally over the top of the machine. The concentrates obtained from this cell were conveniently filtered by allowing the water to drain out through linen bags.

The results given in Table IV of pages 22-25 were obtained for the different samples of graphitic ores that were received from various provinces.

Reference to table IV indicates that only small concentrations of the collectors and frothers should be employed. There has been a difference in opinion regarding the use of sodium silicate as a depressant. Anon (54) finds that the use of sodium silicate as the gangue depressant is harmful while Clemmer and others (50), (55) claim that the addition of sodium silicate is definitely advantageous. Experiments carried out at the Indian Institute of Science indicate that sodium silicate does function as a gangue depressant during flotation of graphite. Only in the case of Mysore amorphous graphite (Expt. 29) the effect of sodium silicate was not noticeable on account of the large quantity of the silicious matter (80-85%) present in the graphitic ore. In certain samples of flaky graphite (Travancore samples, for example) it is beneficial to divide the ground ore into two grades +100 mesh and -100 mesh. The coarse variety (+100 mesh) can be employed in the crucible industry after beneficiation while the fine variety (-100) can be used in the paint and lubricant industries. In general, it has been found that finer grades of the ore contain more gangue material (Expts 23-28) than the coarser ones.

In conclusion it can be stated that the Indian graphitic ores can be beneficiated to a fair degree of purity by the flotation process. The exact method of purification can be determined for each sample of the ore after preliminary laboratory trials. At present it is not economical to beneficiate the amorphous varieties of Indian graphitic ores since it can not compete with the artificial graphite either in quality or in price.

VI PREPARATION OF GRAPHITE LUBRICANTS FROM INDIAN GRAPHITE

Colloidal Graphite was considered to be an essential defence material and owing to supplies from abroad being cut off during the war, it was considered desirable to investigate the immediate possibilities of preparing a lubricant of this nature from purified natural Indian graphite. Colloidal graphite is generally prepared from artificial graphite because of its purity and also of its finely divided condition which eliminates elaborate treatment to bring the particles into a colloidal suspension. The natural amorphous variety which is also very fine would be a suitable starting material provided the purity of the material is brought up to the level of the artificial graphite. India had no artificial graphite industry, nor could the material be imported from abroad during the war years. The only starting material available was, therefore, purified natural graphite of the flaky or crystalline varieties.

Acheson was the first to use graphite in the preparation of lubricants. Later in 1913 Karplus (56) took out a patent for the production of colloidal graphite from natural graphite with the aid of oxidising agents and he used gelatin, albumin and gum arabic for the final stabilisation of the sol. In 1916 de Hae'n (57) employed almost the same method but used saponin as the stabilising agent. In 1930, Hatschek (58) developed a method of masticating amorphous graphite through rolls with a soluble fatty acid soap. The suspension was then coagulated by the addition of metal salts or hydrochloric acid so that the graphite particles were stabilised with a coating of the insoluble fatty acid salt. The coagulum was then transferred to oil. In conjunction with Riedel (59) de Hae'n suggested a method of facilitating fractional sedimentation in a centrifuge with the aid of organic electrolytes like ammonium humate, lignin sulphate etc. Strohbach (60) further claimed the use of aldehydes, ketones and methyl amine as suitable additions to colloidal graphite to render it stable towards heat.

The preparation of colloidal graphite consists mainly of grinding the graphite to a fine state, suspending the ground material in suitable oils or water and finally stabilising the colloid. The deflocculating agents used during grinding are : cellulose sulphate waste liquors, tannin extracts, thickened fatty oils, and polymerised iso-butylene. The various stabilisers used are : petroleum resins, rubber, yolk of egg, ammoniacal solutions of glue and gelatin, tannins, sulphate liquors, glycerides, brown oils, polymerised or condensed lipoids,

phosphatides, rubber dissolved in mineral oil, polymerised or unsaturated hydrocarbons and lecithin.

Experimental

Graphite, purified to 99% and powdered to 200 mesh was mixed with
 Methods tannic acid (6% by wt. of graphite taken) and suspended in dis-
 Employed tilled water and ground in a Premier colloid mill. Grinding in
 a rotary press, an edge runner and in a homogeniser, however, proved ineffec-
 tive. Ammonia was added to prevent flocculation. The coarser particles were
 allowed to settle down into a sediment, by long standing. The supernatant sus-
 pension, was decanted and coagulated with the minimum amount of hydro-
 chloric acid. The major portion of the water was removed in a centrifuge. The
 coagulated graphite was then mixed with lubricating oils (10% of graphite by
 weight). Thorough mixing was effected in a colloid mill and the mixture was
 heated in an oil-bath at about 65°C with constant stirring, when the moisture
 was removed. The addition of a solution of rubber in benzene and aluminium-
 distearate was found to be beneficial.

A second process investigated by Hatschek (58) gave better results. Sodium oleate was used as the dispersing agent in place of tannic acid, and aluminium sulphate was used to coagulate the suspension. The rest of the procedure was the same as above. The addition of rubber solution was found to be beneficial in this case also. The colloid mill having proved inefficient, a small tripple roller mill was employed. The samples thus prepared revealed a series of defects. The ash content of the graphite was very high and the ash constituted entirely of iron with no silica. The graphite content was only 13%. In Acheson's "Oildag", the graphite particles were of the order of 1-2 μ in size while in the locally prepared sample 75% of the particles were of 1-7 μ , 25% of 30-9 μ and only a very small number of 59.5 μ in size. When the sample was diluted with a small amount of petroleum ether, no separation of graphite was observed. Settling however, took place rapidly on diluting with two parts of ether. The sample was passed as being "satisfactory" for defence purposes. Another sample treated with glue showed the following characters on analysis. Graphite content 13% ash content of graphite 5% and the average particles size, 7 μ . This sample showed separation of particles on dilution with two parts of ether while the Acheson product was absolutely stable. The sample was however, considered to compare favourably with Acheson's oildag and it was found satisfactory for lubricating gun slides, cradle guides and breech mechanism. It was found to be unsuitable for mixing with fuel as an upper cylinder lubricant and also as a running-in compound in new engines.

Samples were also prepared by direct mastication of graphite in oil, employing a tripple roller mill. About 1% by weight of rubber solution in benzene was added to powdered graphite together with sufficient amount of lubricating oil form a paste. The pasty mass, after repeated passings through the roller mill was diluted with oil and centrifuged. The sample prepared proved similar to the one treated with glue. Its suitability for use on gun slides, etc., was also confirmed. The sample was still far inferior to the Acheson product and it was thought desirable to employ a roller mill with a large roll diameter.

The following method gave better results. Above 320 gms. of finely powdered crystalline graphite, previously purified by froth flotation, was mixed with a saturated solution of rubber in benzene (3.5% by weight of graphite). To this, about 95 c.c. of B.O.C. 120 B lubricating oil was added and the product was kneaded well into a thoroughly pasty mass. The paste was then passed several times through a tripple roller mill, fabricated at the workshop of the Indian Institute of Science. The tripple roller mill had three chilled rolls, $6\frac{1}{2}$ " in diameter and with adjustable gaps. The central roll slides to and fro transversely while rotating and the third roll had a water jacket, for cooling. Hundred c.c. of B.O.C. 120 B and about 30.c.c. of B.O.C., 50 lubricating oils were added to the paste and the treatment in the roller mill was repeated. The product was finally diluted with lubricating oil in an edge runner and a stabilising agent (crude lecithin) prepared in the laboratory from egg yolk, was added. The final product consisted of a thick thixotropic paste. Portions of this paste were further diluted and centrifuged to remove coarser particles. The sample obtained showed considerable improvement over the former one and was stable towards petroleum ether. The graphite content was 15-18% and had too many particles of size greater than $1-2\mu$. A centrifuge powerful enough to remove these large particles being not available, the suspension was diluted with benzene to facilitate separation and then centrifuged for 44 mins. in a centrifuge with an r.p.m. of 3000. The suspension was decanted and the solvent was removed under vaccum. This product had a graphite content of 7% with uniform distribution of particles of $1-2\mu$ in size. Dilution with petroleum ether showed very little separation even after standing for a fortnight. There was also no deterioration after storage for one year.

In conclusion, it can be stated that the production of a high quality graphite lubricant from amorphous Indian graphites is a comparatively easy process. Even natural crystalline graphite can produce satisfactory products, provided stainless steel roller mills and high power centrifuges are employed.

Summary

TABLE IV

Expt. No.	Graphite sample employed.	Mesh of the ore I. M. M.	Cell used	Frother % on wt. of ore	Collector % on wt. of ore	Depressant % on wt. of ore
1	East Godavari area	-90 to +200	Ruth	Cresylic acid .23*	Cresylic acid as before	...
2	"	"	"	0.46	"	...
3	"	"	"	0.23	"	...
4	"	"	"	0.46	"	...
5	"	"	"	0.23	"	...
6	"	"	"	0.46	"	...
7	Concentrate from Expt. 4	-200	"	0.2	"	...
8	" Expt. 7	"	"	Xanthate	"	...
9	East Godavari area	-90 to +200	Rabone	Cresylic acid 0.23	"	...
10	Travancore Graphite	-100 to +150	Ruth	Turpentine 0.06	Xanthate ethyl 0.02	Na. silicate 0.03-0.15
11	Betul, Central Provinces.	-90 to +200	Ruth	Eucalyptus oil	Xanthate ethyl 0.23	Na. silicate 0.23
12	"	-200	"	"	Xanthate isoamyl 0.22	"
13	Concentrate from 11 and 12	-90	"	"	"	"
14	Concentrate from 13	"	"	Cresylic acid 0.3	"	"
15	Original ore sample	-90 to +200	Rabone	Eucalyptus oil	Xanthate ethyl 0.02	"
16	Concentrate 15, re-ground for 20 min. in edgerunner	...	Ruth	"	"	"
17	Concentrate 16 re-ground wet to -200	-200	"	"	"	"

*After 4 successive flotations, the ash content of the concentrate = 7.6

pH. employed	Ash content of original sample %	Ash content of concen- trate %	Recovery %	Composition of the ore %	Remarks
3.3	36	5.2	90	C = 63.4 Rest = 36.6	Collectors tried: Cresols, turpentine oil, cresylic acid and potassium amyl xanthate
"	"	4.6	"		Condition of the Ore: Crystalline lumps, can easily be crushed
4.3	"	2.8	"		
"	"	2.7	"		
7.0	"	5.7	"		
"	"	4.9	"		
4.3	2.7	1.9	...		
10.0	1.9	1.8	...		
4.3	36.0	4.0	...		
10.0	20.8	4.9	65	C = 79.2 Rest = 20.8	Collectors tried: kerosine, cresylic acid, potassium iso-amyl xanthate. Higher con. of xanthate (0.2%) was ineffective Condition of the ore: lumps of plumbago quality, ground to flakes at 100 mesh. Hence to be used in the crucible industry. Pulp density: 45% solids during conditioning 12% solids while frothing.
9	70	43.8	65	C = 30 Rest = 70	Collectors tried: Cresylic acid, potassium ethyl xanthate and isoamyl xanthate, xanthate found to be the best.
10	70	45.0	"		Condition of ore: crystalline lumps 1"—4" in dia. containing mostly silicious impurities. Addition of Na silicate was advantageous. On account of the poor quality of the ore, the pulp density was 12% solids while frothing.
10	44.5	26.9	"		
4.2	26.9	15.0	...		
9	70	20.5	40		
9	20.5	31.1	88		
9	13.1	10	87.5		

Expt. No.	Graphite sample employed.	Mesh of the ore I. M. M.	Cell used	Frother % on wt. of ore	Collector % on wt. of ore	Depressant % on wt. of ore
18	Oily plumbage from Ceylon	+ 90	Ruth	Eucalyptus oil	Xanthate ethyl 0.02	Na. silicate 0.2
19	"	- 90	"	"	"	"
20	Concentrate from 18	- 90	"	"	"	"
21	FDY powder from Patna State	+ 200	Ruth	Eucalyptus oil	Xanthate ethyl 0.015	Na. silicate 0.02
22	Concentrate from 21	"	"	"	"	0.1
23	FDY powder	- 200	"	"	"	0.02
24	Concentrate from 23	"	"	"	"	0.1
25	"501" lump from Patna State	-40 to +80	Ruth	Eucalyptus oil	Xanthate ethyl 0.02	Na. silicate 0.1
26	Concentrate from 25 reground to -80	-80	"	Turpentine	Kerosine 0.08	Nil
27	Mysore "amorphous"	- 200	Ruth	Eucalyptus	Xanthate 0.02-0.05	Na. silicate 0.2

P. H. employed	Ash content of original sample %	Ash content of concen- trate %	Recovery %	Composition of the ore %	Remarks
9	24.5	7.3	86	C = 75.5	Condition of ore: A second grade quality marketed as "Amorphous" from Ceylon. Powder sample having flakes and fines. Analysis of the sieved samples showed that the % of graphite was higher in +90 than in -90 mesh. This showed that the impurities could be selectively ground.
"	"	13.0	...	Rest = 24.5	
"	7.3	3.0	90		
9	21.9	6.3	94	C = 66.5 Rest = 33.5	Condition of ore: sample was fine powder. (+ 200 mesh = 45.7% and - 200 mesh = 54.3%). Even in this sample concentration to a fair degree is brought about by sieving the sample. Ash content of + 200 mesh = 21.9% - 200 " = 43.5%
"	6.3	3.0	90		
"	43.5	15.0	88		
"	15.6	8.6	85		
9	8.6	6.4	95	C = 88.8	Condition of the ore: small lumps of flaky aggregates of plumbago quality +80 sample has higher carbon content than -80 sample. size 9 analysis - 40 to + 80 = 61% (ash = 8.6%) - 80 = 39% (ash = 15.3).
7	6.4	4.3	93	Rest = 11.2	
9	85.0			C = 15 Rest = 85	Poor quality amorphous variety. Can be used without purification for foundry purposes. Addition of the depressant had no effect on the beneficiation.

VII EQUIPMENT REQUIRED FOR THE BENEFICIATION OF GRAPHITIC ORE

Exact information regarding the details of equipment for the beneficiation of graphite ore can be given after ascertaining the following:

Introduction

(1) The quantity of the ore that has to be beneficiated per day, (2) The nature of the impurities present in the ore; (3) The grindability of the ore; (4) The fineness of the product that is required; (5) Location of the plant. It is obvious that during peace time purified natural graphite cannot compete with artificial graphite in certain industries like the manufacture of lubricants where the graphite must be extremely pure and very finely divided. But in industries where refractoriness is the prime factor (like the crucible industry) purified natural graphite is always preferred. In fact the Alabama Flake Graphite Co., U.S.A., is recovering the flaky graphite for the manufacture of crucibles from an ore which contains only 5-7% graphite (61). But this Company purifies 400 tons of the ore per 24 hours. Another important factor to be taken into account is the quantity of the purified graphite that is required for Indian Industries. In the following calculations, it is assumed that 25 tons of high grade graphitic ore containing 60% graphite has to be purified to give 15 tons of 98% graphite. The accompanying flow sheet illustrates the usual plan for the concentration of graphite by flotation process (61).

Ore from the 25 ton coarse ore bin is fed to the 8" x 10" universal jaw crusher by means of 18" Denver apron ore feeder. The discharge from the crusher is elevated by a belt conveyor to a 18" x 36" single deck vibrating screen with 1" opening screen cloth. The jaw crusher is to be driven by a 15 H.P. motor through a V-belt drive.

Crushing

The screen undersize falls into the fine ore bin. The oversize is taken up by a belt conveyor, running parallel to the conveyor of the jaw crusher, and conveyed to 6' diameter edge runner. The edge runner is to be driven by a 20 h.p. motor through a V to flat belt drive. The discharge from the edge runner falls on to the belt conveyor of the jaw crusher and thus the circuit is completed.

The ore at minus 1" is fed to a 3' x 9' ball mill by means of a Denver adjustable stroke feeder. The ball mill has an air separator enclosed in its circuit. The ball mill is to be driven by a 35 h.p. motor through V-to-flat drive. The ball mill discharges by gravity to a 24" x 36" Denver conditioner where the reagents are added.

Grinding

The product from the conditioner enters a set of 4 cells of No. 12 (22" × 22") Denver "Sub-A" flotation machine to be driven by 2 h.p. motors, each motor driving 2 cells. The rougher concentrates from the last 3 cells enter the first cell and a high grade concentrate is obtained. The contact time in the cell is approximately 8 to 12 minutes.

The final graphite product from the cell is pumped by a 1" vertical sand pump to alternate settling bins with porous false bottoms for drainage. The graphite drains down to about 45-50% moisture.

Dewatered concentrates from the settling bins are transported by a 6" diameter screw conveyor to a small rotary drier encased in brick work and indirectly fired. Any cheap fuel may be used to fire the drier.

The product from the storage hopper will have a particle size of approximately +50 mesh. The coarser graphite may be given a light polishing or grinding treatment in Buhr mills, depending on the quality of the product required.

The approximate operating cost for such a flotation process is given below:

Approximate Capital Cost:

1	Equipment	...	Rs. 100,000
2.	Building	...	" 50,000
3.	Spare parts, etc.	...	" 10,000
			<hr/>
			Rs. 1,60,000
			<hr/>

Approximate operating cost: : per ton of flotation concentrate

Power at 6 pies per unit	...	Rs. 4.2
Chemical	...	" 3.4
Fuel	...	" 6.0
Water	...	" 2.0
Management and Labour—one } Manager and Chief Chemist on } Rs. 500/- p. m. } 2 Assistant Chemists on Rs. 150/- } per month each } 3 Mechanics on Rs. 60/ per month } each } 45 workmen (15 in each shift) on } Rs. 30/- per month each. }	...	" 6.2
Depreciation on equipment	...	" 2.0
Depreciation on Buildings	...	" 1.0
Miscellaneous	...	" 4.0
		<hr/>
Total	...	Rs. 28.8
		<hr/>

The cost of purified graphite will obviously depend on the percentage of graphite in the ore and the fraction recovered.

Following are the prices set up by the U. S. Government for graphite (61)

Grade No.	Mesh size U. S. Standard	Price in cents per lb.	Price in rupees per ton	General Uses
1	+50	13	780	Crucibles
2	+70	11	660	Crucibles and lubricants
3	+100	7	420	Lubricants, electrodes pencils, foundry facings, paints.
4	+100	5	300	Dust for foundry fancings, muni- tions, etc.

The Operating cost for a few minerals is given below *by way of comparison*

	Operating cost per ton of flotation concentrate.
1. Tailings of Measbi washing plants contain- ing 24% Fe and concentrated to 62.0% Fe (62)	Rs. 3/-
2. Tailings from Wright Hargreaves Mines, Ltd., containing 1.4% FeS ₂ and traces of gold to be concentrated to 80% FeS ₂ and gold recovered (63)	Rs. 4-6
3. Net operating cost for floating zinc ore from Mascot, Tenn. assuming 93.3% re- covery (64)	Rs. 27-30
4. Tailings recovered for gold in Columbia (65)	Rs. 10-20

The operating cost of Rs. 29 - per ton worked out in this section is very high since the plant is designed for working of only 25 tons per day. Under very favourable conditions working with 400 tons per day the operating cost may be reduced further. The establishment of a large refining plant depends first on the need for such a large quantity of graphite produced and secondly on the availability of a large quantity of the ore at one

Conclusion

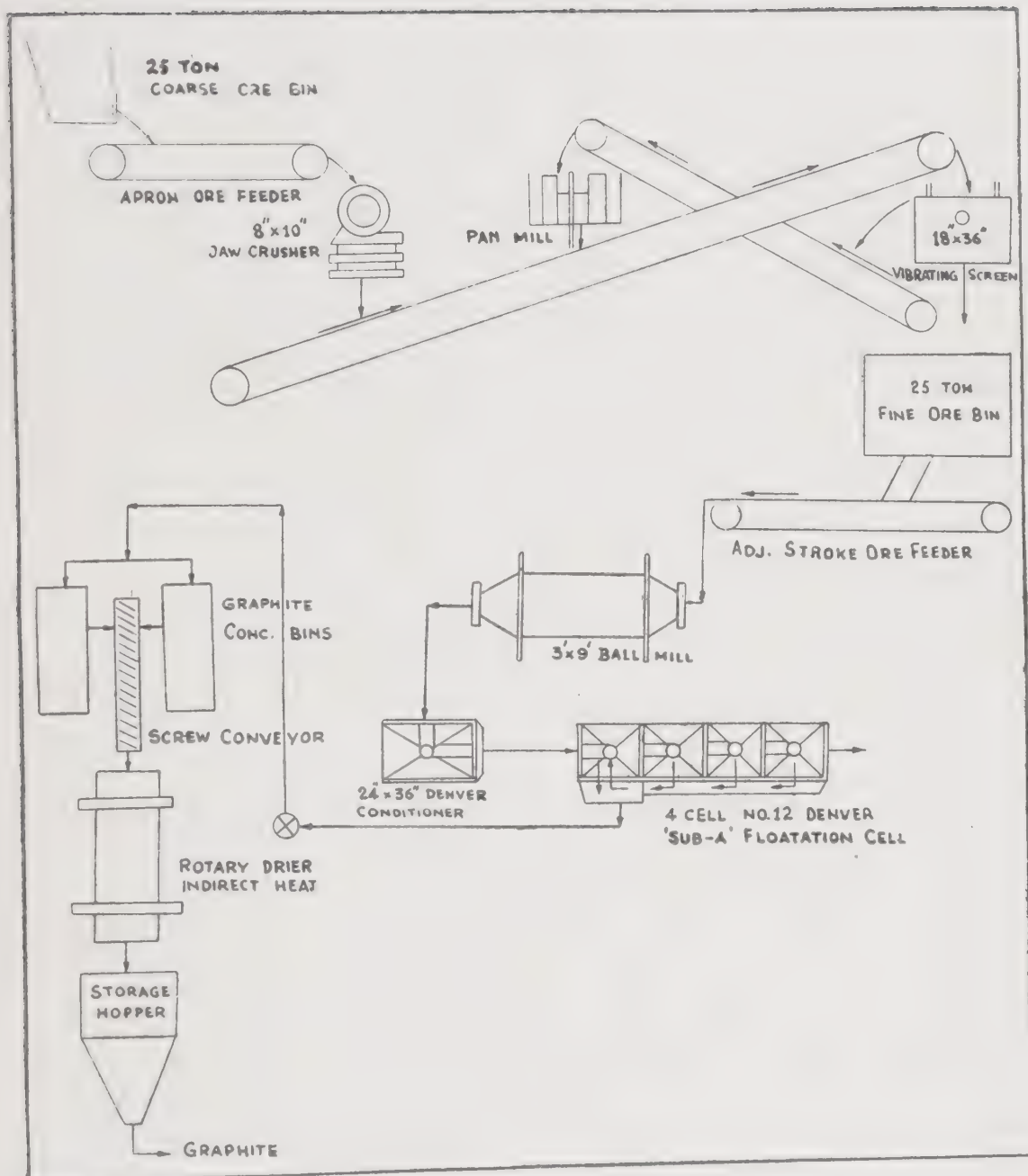
place so as to work out the process of purification for a period of 10-15 years. These two factors should be thoroughly investigated before starting a large scale flotation plant. It is desirable to have a pilot plant for flotation attached to some indigenous graphite industry (crucible industry for example). Although the cost of beneficiation will be of the order of Rs. 29/- per ton, the purified graphite will still be cheaper than the imported one. The cost of graphite ore is generally estimated to be Rs. 5/- per ton at the mines and thus it may be profitable to beneficiate an ore which has 30% graphite since the cost of imported graphite is about Rs. 300 per ton.

REFERENCES

- 1 Sauffrignon, Mines, Carriers, grandes, enterprises May, 1933 (cf. Amer. Chem. Abs. 28 : 6605, 1934)
- 2 Spence, H. S. "Graphite" p. 10. publication by Canada Dept. of Mines.
- 3 Briggs, Proc. Roy. Soc. (Edin) 54 : 121 (1933-34)
- 4 Jodl. Breunststoff 22 : 157 (1941)
- 5 Mentell C.L. "Industrial Carbon" Chapman and Hal, London.
- 6 Donath "Der Graphit" Leipsig.
- 7 Spence H.S., Bulletin on Graphite.
- 8 Draper C.R. "Graphite as paint and pigment" Paint and paint manufr. 12 : 20 (1940)
- 9 Canad. Chem and Process Ind. 27 : 132 (1943)
- 10 Godbole N.N. 'Manufacture of lead and slate pencils' Benares, 1945.
- 11 Contribution to the Dictionary of Economic Products and Industrial Resources of India on 'Graphite' by Dr. M. S. Krishnan, Geological Survey of India.
- 12 Rec. G. S. I., 1935 LXX; 116
- 13 Mem. G.S.I., 1875, XI : 64
- 14 Rec. G.S.I., 1932, LXV : 50
- 15 Rec. G.S.I., 1938, LXXIII : 54
- 16 Mem G.S.I., 1902, XXXIII : 14 part III
- 17 Min. Surv. Reps. Jammu and Kashmir on non-metallic Minerals and on Graphite deposits of Braripura.
- 18 Mem. G.S.I., 1880, XVI : 25
- 19 Rec. Mys. Geol. Dept. XIX : 117 (1920)
- 20 Rec. Mys. Geol. Dept. XVIII : 128 (1919)
- 21 Rec. G.S.I., 1924, LVI : 181
- 22 Rec. G.S.I., 1908, XXXVI : 166
- 23 Rec. G.S.I., 1938, LXXIII : 55
- 24 Rec. G.S.I., 1913, XLIII: 53
- 25 J A. Dunn. 'Indian Mining' 1943, p.19
- 26 Ibid p. 116
- 27 Ibid p. 141

- 28 "Accounts relating to the sea borne trade and navigation of British India".
- 29 Spence H.S. Ref. 2, p.112.
- 30 Bessel and Bessel German Patent No.42, Class 22 (1877)
- 31 Bessel and Bessel German Patent No.39369 (1886)
- 32 Elmore English Patent 21948 (1898)
- 33 Potter Victorian Patent 18775 (1901)
- 34 Froment English Patent 12778 (1902)
- 35 Cattermole Australian Patent 1910 (1904)
- 36 A.F.Taggart 'Handbook of Ore Dressing' John. Willy & Sons, New York (1927)
- 37 I. W. Wark "Principles of Flotation" (1938)
Australasian Institute of Mining and Metallurgy (Inc) Melbourne.
- 38 A.M. Gaudin 'Principles of mineral dressing' (1939)
- 39 "Flotation Fundamentals" Bulletin by Dow Chemical Co.
- 40 E.W. Mayer and H.Schanz 'Flotation' (in German) S. Hirzel, Leipzig 1931.
- 41 P. Rabone, Flotation Plant Practice Mining Publications Ltd., London 1932.
- 42 A.M. Gaudin 'Flotation' McGraw-Hill Book Co., New York 1932.
- 43 I.W. Wark and A.B.Cox J.Phys. Chem. **39**: 551 (1935)
- 44 Del Giudice, Eng. and Mining J. **139** : 291 (1936)
- 45 A.M.Gaudin "Principles of Mineral Dressing" (1939)
- 46 E.Edser. Fourth report on Colloid Chemistry 4: 263 (1922)
- 47 Ref. 45, p 403
- 48 Ref. 37, p. 39
- 49 L.S. Foster, University of Utah. Dept. of Min and Met. Research Tech. paper No. 21927
- 50 Clemmer, Smith etc. Geol. Surv. Alabama. Bull **49** : 101 (1941) Amer. Chem. Abs. **36** : 1146 (1942)
- 51 Gandrud etc. Amer. Chem, Abs. **28** : 2305 (1934)
- 52 Bulletin No. M4-B 34 supplied by the Denver Equipment Co., U.S.A.
- 53 Rabone. "Flotation Plant Practice" (1939) p.109. Mining. publications Ltd., London E.C.2.

- 54 Anon.S.African Min. Eng. J. **52**: 213, 268 (1941)
 - 55 M.A. Matveev Amer. Chem. Abs. **38** : 2592 (1944)
 - 56 H. Karplus, Ger. P. 292729. Nov. 13 (1913)
 - 57 E. De Haen-Swiss, P. 73801 Dec.1, (1916)
 - 58 Britt, P.366128, Oct. 30 (1930)
 - 59 J. D.Riedel—E. De Haen, A. G. Ger. P. 509500 Feb. 15, (1924)
 - 60 E-Stronbach —Austrian—P. 130026 and 130027 July 15, (1931)
 - 61 H.J. Gisler—Deco Trefoil March 1945 page 5.
 - 62 Eng. Min. Journal **140**: 35 April (1939)
 - 63 Eng. Min. Journal **140** : 45 May (1939)
 - 64 Eng. Min. Journal **141** : 39 July (1940)
 - 65 Eng. Min. Journal **143** : 61 March (1942).
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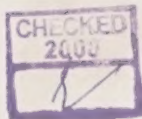
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